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Indian Standard

METHODS OF SAMPLING BENTONITE

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

METHODS OF SAMPLING BENTONITE

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Indian Standard

METHODS OF SAMPLING BENTONITE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 July 1982, after the draft finalized by the Methods of Sampling Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The primary object of sampling of any material is to draw an inference about the quality of the lot on the basis of information derived from the sample. If the consignment is of uniform nature the inference so drawn is almost precise and gives an accurate estimate of the quality, but when the material is heterogeneous in nature as is often the case with any raw material, the method by which sample is obtained becomes critical in inferring about the quality of the consignment.

0.3 The efficiency of sampling largely depends on the degree of homogeneity of the consignment and the size of sample. In case of heterogeneous consignment, it is obvious, that the size of the sample should be larger to arrive at an accurate estimate of the quality.

0.4 For the purpose of sampling, bentonite has been divided into two categories, namely, lumps and powder. Lumps consist of all sizes up to 200 mm. Powder consists of material with maximum particle size of 150 micron.

0.5 For obtaining reliable conclusion it has been recommended that as far as possible bentonite be sampled when in motion, that is, during loading or unloading. When sampling of bentonite from stock piles becomes inevitable, sectional or trench sampling method may be used for stock piles up to a maximum height of 1·5 metres but the representativeness of the sample drawn in this manner cannot be assured and hence the reliability of the conclusions is not assured.

0.6 For the determination of size distribution of bentonite, sieves conforming to IS : 460 (Part I)-1978* or IS : 460 (Part II)-1978† shall be used. When such sieves are not available other equivalent standard sieves as adjudged by the aperture may be used.

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*Specification for test sieves: Part I Wire cloth test sieves (*second revision*).

†Specification for test sieves: Part II Perforated plate test sieves (*second revision*).

0.7 In reporting results of test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the methods of sampling of bentonite from wagons/trucks, ships stock piles and bags for the determination of size distribution, moisture content physical and chemical characteristics of the material in the lot. It also includes the method for reporting the quality of the material sampled.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Lump — Bentonite of size up to 200 mm.

2.2 Powder — Bentonite with maximum particle size 150 microns.

NOTE — The maximum particle size is the size of the aperture of the sieve on which approximately 5 percent of the material is retained.

2.3 Lot — The quantity of bentonite indicated to be of the same category and offered for inspection at one time. A lot may consist of whole or part of the quantity ordered for.

2.4 Sub-lot — The quantity of bentonite in each of the parts into which a lot is divided for the purpose of sampling.

2.5 Increment — The quantity of bentonite obtained by a sampling device at one time from a lot or sub-lot. The quantity of a single increment is called size of increment.

2.6 Unit Sample — The quantity of bentonite collected at one point in sectional sampling.

2.7 Gross Sample — Sample as collected from a sub-lot, that is, the quantity of bentonite of several increments of unit samples taken from a sub-lot.

2.8 Size Sample — Sample taken for determination of particle size distribution of the lot or sub-lot.

2.9 Laboratory Sample — The quantity of bentonite obtained by reducing a gross sample following the specified procedure and indicated for laboratory tests.

*Rules for rounding off numerical values.

2.10 Moisture Sample — The sample to be used for determination of moisture content of the lot or sub-lot.

2.11 Composite Sample — The quantity of bentonite obtained by mixing together proportionate quantities of material from each of the laboratory samples representing the sub-lot into which a lot has been divided.

3. METHODS OF SAMPLING

3.1 Division of Lot into Sub-lots — For the purpose of sampling a lot shall be divided into a number of sub-lots of approximately equal mass as specified in Table 1.

NOTE — When it is not possible to have sub-lots of approximately equal masses, sub-lots of varying masses may also be permitted.

TABLE 1 MINIMUM NUMBER OF SUB-LOTS INTO WHICH A LOT IS TO BE DIVIDED

(*Clauses* 3.1, 4.1.1, 4.2.1, 4.3.1 *and* 4.4.2)

MASS OF THE LOT (IN TONNES)	MINIMUM NUMEER OF SUB-LOTS
(1)	(2)
Up to 1 500	2
1 501 „ 3 000	3
3 001 „ 5 000	5
5 001 „ 7 000	7
7 001 „ 10 000	8
10 001 and above	10

3.1.1 Representative gross sample shall be drawn from each of the sub-lots and shall be kept separately. Thus there will be as many gross samples as the number of sub-lots into which the lot has been divided.

3.2 The number of increments to be taken from a sub-lot for making the gross sample shall be governed by the mass of the gross sample and the mass of the increment as specified in Table 2 for the two categories of the bentonite. These increments shall be evenly distributed over sub-lots. The increment shall be drawn with a suitable sampling scoop (*see* Fig. 1) at regular intervals.

4. VARIOUS SAMPLING METHODS

4.1 Sampling from Wagons/Trucks

4.1.1 *Sub-lots* — For the purpose of sampling all the wagons/trucks in a lot shall be divided into a suitable number of sub-lots of approximately equal mass in accordance with the requirements of Table 1.

TABLE 2 MINIMUM MASS OF THE GROSS SAMPLE AND MINIMUM NUMBER OF INCREMENTS

(*Clauses* 3.2, 4.1.2.1, 4.2.2, 4.4.2 *and* 4.4.5)

CATEGORY OF BENTONITE	MINIMUM MASS OF GROSS SAMPLE (kg)	MASS OF INCREMENT, APPROXIMATE (kg)	MINIMUM NUMBER OF INCREMENTS
(1)	(2)	(3)	(4)
Lumps	120	4	30
Powder	25	1	25

4.1.1.1 A representative gross sample shall be drawn from each of the sub-lot and shall be kept separately. Thus there will be as many gross sample as the number of sub-lots into which a lot has been divided.

4.1.2 In order to get representative gross sample, the bentonite shall be sampled, as far as possible, in a steady motion during loading or unloading of the wagons/trucks.

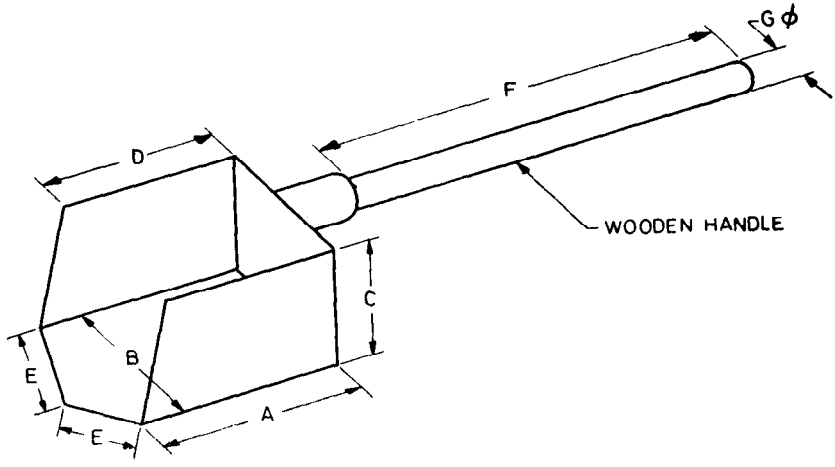
4.1.2.1 A minimum of 50 percent of the wagons/trucks shall be selected at random from the sub-lot. The total number of increments to be taken from the selected wagons/trucks, the mass of the increments and the gross sample shall be in accordance with Table 2. Approximately equal number of increments shall be taken from each of the selected wagon/truck. The increments to be taken within a wagon/truck shall be evenly distributed. These increments shall be drawn with the help of a suitable sampling scoop (*see* Fig. 1) at regular intervals at the time of loading or unloading of the wagons/trucks.

NOTE — While taking the increments as prescribed above, if a large lump is encountered at any point and cannot be taken in the scoop, it shall be picked up as such. Such lumps shall be accounted for in the determination of size distribution as given in **5** and in the reduction of gross sample as given in **6**.

4.1.3 *Sampling From Loaded Wagons/Trucks* — As far as possible sampling from loaded wagons or trucks shall be avoided If it is inevitable, it may be carried out by sectional sampling method as described below. The number of unit samples to be collected from each sub-lot by sectional sampling method will be minimum 10 in the case of lumps and 5 in the case of powders.

For collecting these unit samples, required number of points shall be located at random on the surface of each of the wagons/trucks in the sub-lot. At every selected point a unit sample shall be collected by taking the whole section of bentonite from top to bottom over the area of a circle of 20 cm diameter for lumps and 15 cm diameter for powder. For doing so, the material from the surface up to a depth of approximately 45 cm shall be collected at first. The bottom of the hole so formed shall then be

covered by a plate and the material lying on the sides shall be removed up to that plate so that when the hole is dug further, the material from the sides does not fill up the hole by falling down. This procedure is repeated till the bottom is reached.



Capacity of the Scoop	Dimensions of the Sampling Scoop (in mm)						
(1)	*A* (2)	*B* (3)	*C* (4)	*D* (5)	*E* (6)	*F* (7)	*G* (8)
4 kg (Lumps)	250	250	110	220	160	440	40
1 kg (Powder)	150	150	75	130	100	350	30

Fig. 1 Sampling Scoop

4.2 Sampling from Shipholds

4.2.1 For the purpose of sampling, the quantity of bentonite to be loaded into ships shall be divided into a suitable number of sub-lots of approximately equal masses in accordance with Table 1. From each sub-lot a representative gross sample shall be taken. Thus, there will be as many gross samples as the number of sub-lots into which a lot has been divided.

4.2.2 Sampling shall be carried out only when the material is in motion. Accordingly, at the time of loading or unloading gross samples from each sub-lot may be collected by taking increments at equal intervals of time or quantity. The number and size of the increments shall be as given in Table 2.

4.3 Sampling from Bags

4.3.1 Normally bentonite is packed in jute bags having a capacity of about 50 kg and a lot will consist of a number of bags. These bags will be grouped into a number of sub-lots as per Table 1. If we denote by N the number of bags in the sub-lot and by n the number of bags to be selected for the sample, the gross sample will consist of n increments at the rate of one increment per bag. The number of bags to be selected from each sub-lot is given in Table 3. The weight of each increment will be 4 kg in case of lumps and 1 kg in case of powders.

TABLE 3 NUMBER OF BAGS TO BE SELECTED FOR SAMPLING FROM A SUB-LOT OF BENTONITE PACKED IN BAGS

Size of the Sub-lot in Tonnes	Minimum Number of Bags to be Selected (n)
(1)	(2)
Up to 50	30
51 ,, 100	40
101 ,, 200	50
201 ,, 400	60
401 ,, 800	80
801 and above	100

4.3.2 The bags shall be selected at random from the sub-lot during the shifting of the bags so that one can have easy access to all bags in the sub-lot. The counting of bags is to be done along with shifting operations. Starting with any bag at random, the bags shall be selected at equal intervals of $\frac{N}{n}$.

NOTE — The procedure in Table 3 is based on 50 kg capacity bag. Bags with capacity substantially different from 50 kg shall be sampled by multiplying the figures given in col 2 by the factor K, where $K = \frac{50}{\text{actual capacity of the bag}}$.

4.3.3 Each bag so selected shall be opened and the contents emptied on a non-absorbing surface. The material shall be spread evenly. From this one increment shall be drawn with the help of the prescribed sampling scoop.

4.4 Sampling from Stock Piles

4.4.1 Sampling of bentonite in stationary stock piles shall be avoided. The proper method of sampling is to collect the increment when the stock pile is being formed or dismantled.

4.4.2 *Sub-lots* — For the purpose of sampling, the quantity of bentonite in a stock pile shall be divided into a number of sub-lots of approximately equal mass as specified in Table 1 and the number of increments to be drawn from each sub-lot shall be according to Table 2. From each sub-lot one gross sample shall be taken.

4.4.3 When a stock pile is in process of being built, or shifted, increments may be taken at regular intervals during the process in order that the bentonite in the entire stock pile is covered by sampling.

4.4.4 *Stationary Stock Piles* — When it becomes absolutely necessary to sample a stationary stock pile, sectional sampling (*see* **4.1.3**) or trench sampling method (*see* **4.4.5**) may be used for a stock piles up to maximum height of about 1·5 metres. In the case of sectional sampling, the requisite number of points shall be located at random on the entire surface of the sub-lot.

4.4.5 *Trench Sampling* — The gross samples shall be made up of the number of increments as specified in Table 2. These increments shall be collected as indicated below:

Along a randomly chosen line of bentonite surface of the sub-lot, the trench shall be cut right down to the ground level leaving about 30 cm walking space at the ground level. From the trenches so cut the required number of increments shall be collected with the help of a suitable sampling scoop (*see* Fig. 1) at various points selected as far as possible at regular intervals on both walls to cover the entire length of the trench. In case of larger stock piles, in addition to the trenches the sides of the piles may also be opened to expose the bentonite at places where the trench does not expose the bentonite inside. The increments can be drawn from the sides of the exposed surface.

5. DETERMINATION OF SIZE DISTRIBUTION

5.1 When the gross sample from each sub-lot has been collected in the above manner, it shall be first subjected to size determination using suitable sieves. The size distribution of bentonite in lump form in a lot shall normally be estimated as recommended below:

a) Over 200 mm,

b) Over 100 mm and up to 200 mm,

c) Over 10 mm and up to 100 mm, and

d) 10 mm and below.

The distribution of other sizes for bentonite may be estimated in a similar way.

NOTE — In the determination of size distribution IS sieves of suitable sizes as specified in IS : 460 (Part I)-1978* or IS : 460 (Part II)-1978† shall be used.

5.2 Size Distribution Made on Gross Samples Obtained from Aggregating Unit Samples

5.2.1 Each gross sample shall be screened through selected IS Sieves, the material retained on each of the sieves and that passing through smallest sieves shall be weighed separately. Size distribution of the lot shall be estimated as follows:

a) Over 200 mm, percent $= \frac{a_1 + a_2 + \ldots\ldots}{w_1 + w_2 + \ldots\ldots} \times 100$

b) Over 100 mm and up to 200 mm, percent $= \frac{b_1 + b_2 + \ldots\ldots}{w_1 + w_2 + \ldots\ldots} \times 100$

c) Over 10 mm and up to 100 mm, percent $= \frac{c_1 + c_2 + \ldots\ldots}{w_1 + w_2 + \ldots\ldots} \times 100$

d) 10 mm and below, percent $= \frac{d_1 + d_2 + \ldots\ldots}{w_1' + w_2 + \ldots\ldots} \times 100$

where

$w_1, w_2, \ldots\ldots$ are the masses of the different gross samples;

$a_1, a_2, \ldots\ldots$ are the corresponding masses of bentonite, over 200 mm in size;

$b_1, b_2, \ldots\ldots$ are the corresponding masses of sizes over 100 mm and up to 200 mm;

$c_1, c_2, \ldots\ldots$ are the corresponding masses of sizes over 10 mm and up to 100 mm; and

$d_1, d_2, \ldots\ldots$ are the corresponding masses of sizes 10 mm and below.

5.3 Gross Samples Obtained by Aggregating Increments

5.3.1 Where gross samples do not contain any lumps, the procedure detailed in **5.2** shall be followed for estimating the ore size distribution in the lot, the percentage of ore over 200 mm being nil.

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*Specification for test sieves: Part I Wire cloth test sieves (*second revision*).

†Specification for test sieves: Part II Perforated plate test sieves (*second revision*).

5.3.2 Where the gross samples contain some lumps (*see* Note under **4.1.2.1**) the portion of the gross samples excluding the lumps shall be treated as in **5.3.1.**

5.3.2.1 The size distribution of the lot shall be estimated as follows:

a) Over 200 mm, percent $= \frac{(n \times 4)}{(n \times 4) + w'_1 + w'_2 + \ldots\ldots} \times 100$

b) Over 100 mm and up to 200 mm, percent $= \frac{b_1 + b_2 + \ldots\ldots\ldots\ldots}{(n \times 4) + w'_1 + w'_2 + \ldots\ldots} \times 100$

c) Over 10 mm and up to 100 mm, percent $= \frac{c_1 + c_2 + \ldots\ldots\ldots\ldots}{(n \times 4) + w'_1 + w'_2 + \ldots\ldots} \times 100$

d) 10 mm and below, percent $= \frac{d_1 + d_2 + \ldots\ldots\ldots\ldots}{(n \times 4) + w'_1 + w'_2 + \ldots\ldots} \times 100$

where

$w'_1, w'_2, \ldots\ldots\ldots\ldots$ are the masses of the different gross samples excluding the lumps,

n is the total number of lumps in all the gross samples, and

$b_1, b_2 \ldots\ldots c_1, c_2 \ldots\ldots d_1, d_2 \ldots\ldots$ are as defined in **5.2.1.**

NOTE — Each lump is supposed to represent one increment of 4 kg.

6. REDUCTION OF GROSS SAMPLE

6.1 Moisture Sample — Each of the gross samples shall be sent first for size determination. Only after this, sample preparation shall be conducted for obtaining moisture and laboratory samples. For moisture determination, moisture sample of 20 kg shall be taken from each gross sample in not less than 5 increments after crushing the bentonite to −10 mm by mechanical or manual means. From this 20 kg material, 2 or more samples of 1 kg shall be drawn for moisture determination.

6.1.1 If needed, separate moisture samples may be drawn from the sub-lot. In the case of bentonite of lump category, 5 increments of 4 kg each may be separately taken from various parts of the sub-lot for the purpose of moisture determination. This should be crushed to −10 mm size and out of the crushed material, 2 moisture samples of 1 kg each shall be selected by increment division method. In the case of powder, 5 increments of 1 kg each shall be taken and from this, 2 moisture samples of 200 g each may be collected by increment division method.

NOTE — It has to be borne in mind that such sample may not be representative and may lead to biased results.

6.2 Preparation of Sample for Physical Tests and Chemical Analysis of Bentonite in Lump Form — Bentonite from each gross sample shall be first crushed in a jew-crusher or roll-crusher or by manual method using a hammer, a pounder and a steel plate, till the material reaches — 10 mm size. After collecting the moisture sample as in **6.1**, the remaining material shall be mixed well and reduced to minimum 30 kg which shall then be further processed in stages as detailed in Fig. 2 in order to prepare the laboratory samples for physical and chemical analysis.

NOTE — The lumps obtained while drawing the increments, shall be crushed separately and one scoopful for each lump, approximately 4 kg shall be taken along with bentonite of other sizes in the gross sample for reduction as detailed in **6.2**.

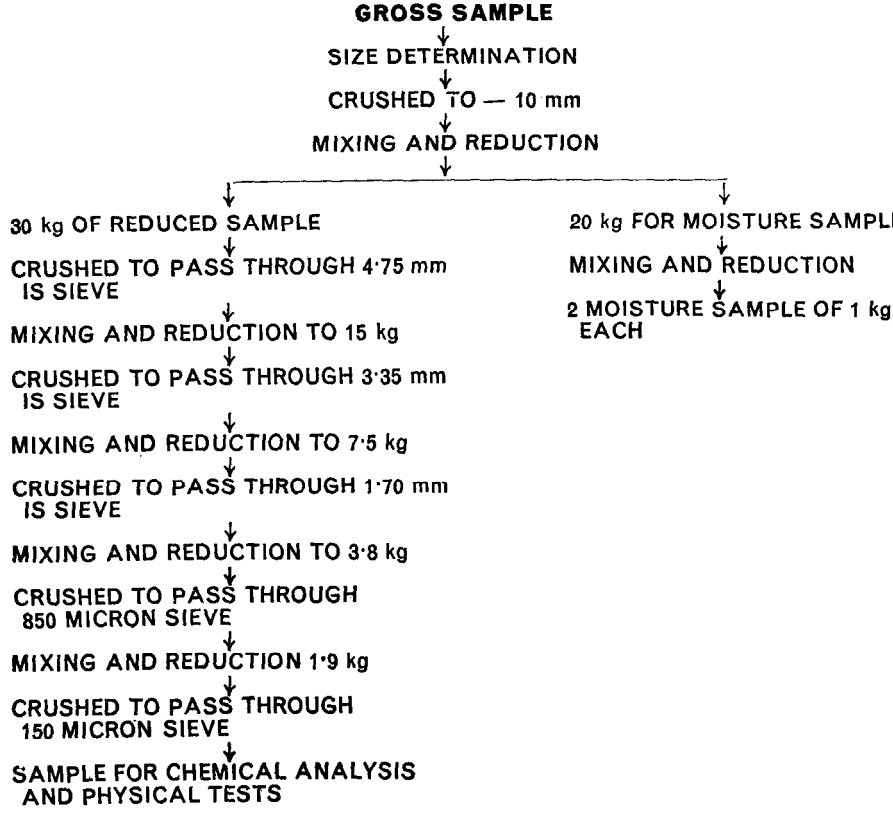

FIG. 2 FLOW CHART FOR PREPARATION OF QUALITY SAMPLES OF BENTONITE OF LUMP CATEGORY

6.2.1 For bentonite in powder form, laboratory samples may be prepared by increment reduction method.

7. NUMBER OF TESTS

7.1 All the moisture samples prepared from different gross samples or drawn separately shall be tested individually for moisture content. If needed, moisture determination may be carried out in duplicate for each gross sample. To avoid any change in the moisture content, the moisture determination shall be carried out as quickly as possible.

7.2 Laboratory Samples for Physical and Chemical Analysis — All the laboratory samples shall be tested individually for important characteristics. For remaining characteristics a composite sample prepared by mixing quantities of laboratory sample proportional to weight of each of the sub-lot, shall be analysed. Unless otherwise agreed to between the purchaser and the supplier the following schedule of testing should be followed:

Characteristics

For which individual laboratory sample to be analysed	For which a Composite sample is to be analysed
a) Gel formation index in case of Type 1 & 2* bentonite	All the remaining characteristics
b) Swelling power for Type 1	
c) Filtration loss in case of Type 3*	
d) Loss of ignition percent for bentonite for rubber industry	

7.3 The sample required for yield test for Type 3 bentonite shall be independently collected from different portions of the lot and one test shall be carried out for yield for every lot.

8. REPORTING

8.1 Reporting of Moisture Content — The result obtained from ith sub-lot shall be denoted by x_i. In case duplicate test results are obtained from ith sub-lot, average of these two results shall be denoted as x_i. The

**See* IS : 6186-1971 Specification for bentonite.

average moisture content of bentonite in a lot shall be calculated as below:

$$\text{Average moisture content} = \frac{\Sigma (m_i x_i)}{\Sigma (m_i)}$$

where

m_i = the mass of the ith sub-lot.

The symbol Σ stands for summation over all i's.

8.2 Reporting of Physical and Chemical Characteristics

8.2.1 For these characteristics, where a composite sample has been tested, only one test will be available and that result shall be reported as the value of the characteristics for the lot sampled.

8.2.2 When only one laboratory sample has been analysed for a characteristic it shall be reported as the value of that characteristic for the lot sampled. When two laboratory samples have been analysed individually from a lot the average of the two test results, shall be reported as the value of the characteristics for the lot sampled. In case if the masses of the two sub-lots differ substantially, the weighted average of the two resutls $\frac{(m_1 x_1 + m_2 x_2)}{m_1 + m_2}$ shall be taken as the value for the lot. The individual results shall be reported to give an indication of the range of variation in quality.

8.2.3 When three or more laboratory samples from a lot have been analysed individually with reference to any characteristic, the following procedure shall be followed to assess average quality of the lots and its limits of variation:

Let x_1, x_2, x_3, ... x_n be the results of analysing n laboratory samples for a particular characteristic.

Calculate,

Average ($\bar{x}$) = $\frac{x_1 + x_2 + \ldots\ldots + x_n}{n}$ if n sub-lots are of approximately equal mass,

or weighted average ($\bar{x}$) = $\frac{(m_1 x_1 + m_2 x_2 + \ldots\ldots + m_n x_n)}{m_1 + m_2 \ldots\ldots + m_n}$

if m_1, m_2 ... m_n are the masses of the n sub-lots;

and Range (R) = the difference between the maximum and the minimum of the values (to be used when n is less than 10)

or ($\bar{R}$) = the average value of ranges. When the number of sub-lots (n) is equal to 10, the corresponding results of n laboratory samples (10) should be constituted into groups of 5 in the order of their occurrence. For each group range (R) should be calculated and the average value ($\bar{R}$) of the R's should be used in the subsequent clause.

The average level of the characteristic in the lot shall be reported as equal to ($\bar{x}$).

The limits of variation in the average level of the lot shall be reported as ($\bar{x} \pm hR$) or ($\bar{x} \pm h\bar{R}$) where h is a factor, the value of which depends on the number of samples analysed. The appropriate value of the factor h is given below:

Number of Laboratory Samples Analyzed	*Value of Factory h*
3	1·30
5	0·51
7	0·33
8	0·29
10	0·31

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	Telegrams : Manaksanstha (Common to all Offices Telephone
Central Laboratory :	
Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010	8-77 00 32
Regional Offices:	
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95
Branch Offices::	
'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003	55 40 21
Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 0
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	8-71 19 9
53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20 10 83
E-52, Chitaranjan Marg, C- Scheme, JAIPUR 302001	37 29 25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Patliputra Industrial Estate, PATNA 800013	26 23 05
Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005	32 36 35
T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	6 21 17

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*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, CALCUTTA 700072	27 10 85
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309 65 28
‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002	222 39 71

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